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Determining % Proliss 100 in Keratin Mousse Using Iodine Solution Method

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Abstract:

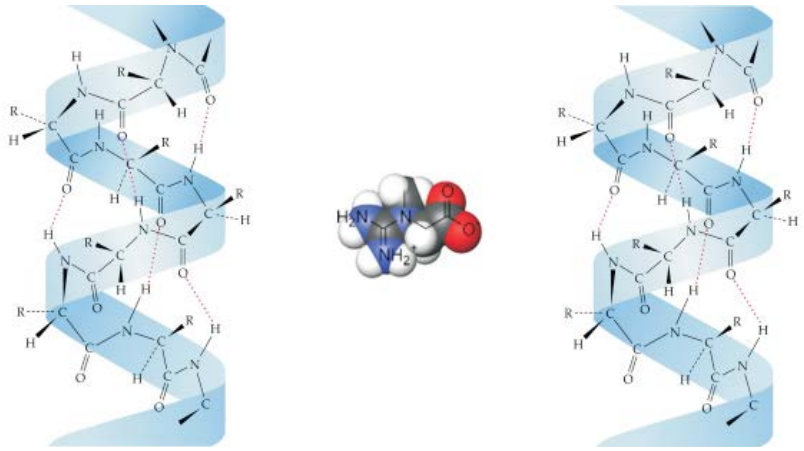
The objective of this experiment is to quantitatively determine the amount of active raw material Proliss 100 manufactured and supplied by a formulation company. The goal is to reproduce the percent of Proliss in Keratin Mousse production sample provided to consumers and provide measurements to be used to assess the accuracy of production with comparison to provided percent formulation for each batch. An iodine solution titration method was performed for this experiment due to the reduction-oxidation reactivity of the Proliss 100. This experiment is still undergoing further testing due to the confidentiality agreement between the manufacturer and formulator.

Introduction:

Proliss 100 is an exclusive technology conceived to be used in capillary formulas based on the acidic oxoacetamides synergy. Due to the association of the oxoacetamides, the carbocysteine and keratin amino acids that make up the Proliss 100 allow a temporary change of the hair structure, providing benefits to hair such as smoother and easier to comb hair, volume control and frizz reduction, and gives a shine and natural look. Keratin Mousse is a product design for consumers such as licensed cosmetologist to aide in the temporary altering of hair structure. The active ingredient that allows this change in the keratin mousse is Proliss 100. The preferred amount of Proliss 100 in consumer products is between 5-25 wt. %, and more preferably 10-20 wt. % based on total weight of the finished composition. The Proliss 100 technical information from the manufacturer's certificate of analysis (COA) includes:

- Aspect (25ºC) : Liquid to lightly opalescent
- Color: Yellow to dark chestnut
- Density (20ºC): 1.270 – 1.370
- pH (sol. 20%): 1.0 – 2.0
- % Active (Refraction): 45 – 55

In some instances most people use relaxers to chemically remove their natural curl pattern, temporarily changing the hair structure, which is an irreversible process. Relaxers are caustic and typically have a pH range of 12 to 14. An alternative process to relaxers that involves removal of natural curl patterns is known as the Keratin treatment system which is classified as a reduction-oxidation process. Commercial redox formulations typically reduce the disulfide bonds in hair with thiol-salts usually ammonia thioglycolate. Reduce hair is mechanically straighten with combing and then oxidized with hydrogen peroxide or sodium bromide to restore disulfide bonds in a new straightened configuration usually lasting 3-4 shampoos. Hair type 4 is more successfully straighten with a relaxer where hair types 2 and 3 are more resistant.



Present enhanced hair straightening system is directed to enhancing the removal of natural curl from hair. Most people of African descent have naturally curly hair unlike people of Caucasian descent, ranging from a wavy to tightly curled configuration. There is a chart that most people, especially hair stylists, follow known as the Global Hair Texture Charting System that will explain the different hair types among women of all ethnicities. This chart was created by an Emmy award winning stylist named Andre Walker best known for his work with Oprah. (reference). He created a hair chart that would be a base for how most women especially of color identify their hair texture. Walker's hair chart had four variations of texture from straight to kinky, Type 1 through 4. This chart has become so identifiable in the hair world that it has become almost infamous within the black hair community today. Now beyond Type 1 (straight hair) there is Type 2A-C (wavy hair), Type 3A-C (curly hair), Type 4A-C (coily/kinky hair), which better defines the variations in textures.

Hair can be classified in various curl patterns:

- Type 1 –straight hair no curl
- Type 2- wavy "S" shaped curls
- Type 3- curly well defined loopy "S" shaped wave pattern
- Type 4- kinky or very tightly curled hair sometimes known as coily hair

Type 3 and 4 have been known to be the most difficult to comb and style because of the bulky volume especially when humid. Sub-types range from letters A-C being thickness (diameter)

- A is fine thickness
- B is medium thickness
- C is coarse (super) thickness

A keratin treatment is applied to the hair in a multi-step process which will activate the Proliss 100 and allow the hair structure to change. The process involves:

1. Wash the hair with an anti-residual shampoo
2. Rinse off the hair till all shampoo is removed
3. Remove water excess with a towel
4. Apply the product with a brush, lock by lock from root to tips
5. Let the product act for 15 minutes
6. Dry the hair with a blow dryer and a rounded brush in order to straight the hair
7. Flatten the hair with an iron at a temperature of 200^{o}C
8. Wait for 15 minutes and then wash the hair with a shampoo and conditioner, let the conditioner act for 3 minutes.
9. Remove water excess with a towel
10. Dry the hair with a blow-dryer
11. Flatten the hair with an iron at a temperature of 200^{o}C

The method chosen for analysis as an iodine solution titration method. When reducing analyte is titrated directly with iodine to produce iodide I- this is called iodimetry. In this process, an oxidizing agent is added to excess I- to produce iodine, which is then titrated with standard thiosulfate solution. Molecular iodine is only soluble in water, but its solubility is enhanced by complexation with iodide, forming triiodide, a brown solution

$$I_2(aq) + I^- \leftrightarrow I_3^-$$

Starch is used as an indicator for iodine. With I_3- titration, starch can be added at the beginning. First drop of excess I_3^- after the equivalence causes the solution to turn dark blue. Triiodide is usually prepared by dissolving solid I_2 in excess KI and must be used immediately or will be oxidized by air. The oxidation reaction of Proliss is similar to Vitamin C.

$$\text{Ascorbic acid} + H_2O \leftrightarrow \text{dehydroascorbic acid} + 2H^+ + 2e^-$$

The method used for determining amount of Proliss % in keratin mousse solution is done by redox titration using iodine. As iodine is added, the Proliss is being oxidized while the iodine is reduced to iodide ions. A simplified equation is presented here for the oxidation of Proliss 100

$$\text{Proliss 100} + I_2 \rightarrow 2\ I^- + \text{oxidized Proliss 100}$$

Iodine formed is reducible to iodide as long as Proliss is present. Iodine is insoluble but can be improved by complexing iodine with iodide to form triiodide.

Experimental Methods:

Materials:

Samples that were used were prepared by Research and Development Formulation Chemist. The two Keratin Mousse Samples were prepared from 14% and 19.5% formulations The reagents that were used were Iodine solution Purchased called Lugol's Diluted Reagent (2% I_2), iodine solution prepared (potassium iodide (KI), potassium iodate (KIO_3) and 3M sulfuric acid (H_2SO_4)), and 1% starch as an indicator. The iodine solution method was performed by standard titration using a 50mL buret.

Procedure:

Procedure:

1. 1% starch indicator provided
2. Pro-liss 100 Standard Solution
    - Weigh 0.25g Proliss 100 standard solution
    - Dissolve in 100mL DI water
    - Dilute to 250mLwith DI water in volumetric flask
      Conc=1mg/mL
3. Iodine Solution
    - Dissolve 5g Potassium Iodide (KI) and Dissolve 0.268g Potassium Iodate (KIO_3) in 200mL DI water
    - Add 3M sulfuric acid (8.8272g in 30mL DI water)
      Calculation: (3mol/L) x (98.08g/1mol) x (1L/1000mL) = 0.29424g/mL
      (0.29424g/mL) x (30mL) = 8.8272g
    - Pour solution into 500mL graduated cylinder
    - Dilute to final volume 500mL with DI water
    - Mix, transfer to 600mL beaker and label
4. Standardize Solution
    - Add 25mL Proliss 100 standard solution prepared in step #2 to 125mL Erlenmeyer Flask
    - Add 10 drops of 1% starch indicator
    - Rinse buret with small amount of iodine solution prepared in step #3 and fill
    - Titrate to endpoint
    - Record volume
5. Keratin Mousse Production Sample
    - Add 25mL of keratin mousse to 125mL Erlenmeyer Flask
    - Titrate to endpoint with iodine solution

Note: 1%=1g/100mL or 10g/L

Calculations: (ml of iodine solution 1/g Proliss 100) = (ml of iodine solution 2/ml Proliss 100)

Iodine solution 1 = volume required to titrate Proliss 100 standard solution to endpoint

Iodine solution 2 = volume required to titrate Keratin Mousse solution to endpoint

14% Keratin Mousse (50% active =7%)

(0.1mL/0.27g) = (0.2mL/X mL Proliss 100 in Keratin Mousse)

0.37x = 0.2

X = 1.85g Proliss in Keratin Mousse

So 25ml Aliquot:

1.85g / 25mL = 0.074g/mL

%= (0.074g/mL) x (100mL/1g) = 7.4% Proliss in 14% (50% active= 7%) Keratin Mousse sample for 100mL sample

So if production sample volume is 250mL

7.4g/100mL) = Xg

19.5% Keratin Mousse (50% active =9.75%)

(0.1mL/0.27g) = (0.3mL/X mL Proliss 100 in Keratin Mousse)

0.37x = 0.3

X = .81g Proliss in Keratin Mousse

So 25ml Aliquot:

.81g / 25mL = 0.074g/mL

%= (0.074g/mL) x (100mL/1g) = 7.4% Proliss in 14% (50% active= 7%) Keratin Mousse sample

% error = (7.4%-7%) – 7% = 5.71% error

1% starch indicator (provided)

▶ Prepared Iodine Solution:

1. Dissolve 5g of Potassium Iodide (KI) and 0.268g Potassium Iodate (KIO3) in 200ml of DI water.
2. Add 30ml of 3M Sulfuric Acid (concentrated)
3. Pour solution into 500ml graduated cylinder and dilute to final volume of 500ml with DI water, mix, transfer to 600ml beaker and label

▶ Proliss 100 standard solutions

1. 250mg weighed out and dissolved in 100ml of DI water
2. Dilute to 250ml with DI water in volumetric flask

▶ Iodine solution: 2 solutions (1 prepared and 1 provided)

▶ Standardized solutions

1. Add 25ml of Proliss Standard solution to 125ml Erlenmeyer Flask
2. Add 10 drops of 1% starch indicator provided
3. Rinse and fill burette with iodine solution
4. Titrate to end point (blue color persisting longer than 20 second)
5. Record volume

▶ Titrate production sample with iodine solution

1. Add 25ml to 125ml Erlenmeyer Flask
2. Titrate to end point color change to starch blue

▶ Standardized solutions

1. Add 25ml of Proliss 100 Standard solution to 125ml Erlenmeyer Flask
2. Add 10 drops of 1% starch indicator provided
3. Rinse and fill burette with iodine solution
4. Titrate to end point (blue color persisting longer than 20 second)
5. Record volume

▶ Titrate production sample with iodine solution

1. Add 25ml to 125ml Erlenmeyer Flask

2. Titrate to end point

The Global Hair Texture Charting System:

Calculations:

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Other calculations include determining the amount of carbocysteine present in Proliss assuming that it was 100% carbocysteine. In this experiment stoichiometry was depended upon being a 1 to 1 ratio in the redox reaction

So I_3- + carbocysteine = oxidized carbocysteine

In order to determine percent carbocysteine, convert the mL by multiplying by 2% reagent into moles of carbocysteine into grams of carbocysteine and then into wt/wt percent.

Calculation as follows:

Example:

Lugol's Iodine Solution or Iodine solution prepared is 2%

2%=2g / 100 mL of solution

2g I_2 / 100ml solution x Density (assuming 1g/1mL) x 0.1 mL = 0.002g I_2

0.002g I_2 x 1mol/253.81 g = 7.88 x 10^{-6} moles of I_2 = 7.88 x 10^{-6} moles of carbocysteine

7.88 x 10^{-6} moles of carbocysteine x 179.191g/mole carbocysteine = 0.00141 g of carbocysteine

0.00141 g of carbocysteine in 25 mL aliquot * 0.27g/ 250 mL stock solution Proliss 100 sample

(0.00141 g of carbocysteine /25mL/ 0.27g /250 mL Proliss sample) x 100 = % carbocysteine in Proliss 100 assuming all the active ingredient is carbocysteine in Proliss

Calculations were performed for remaining Proliss sample weights and aliquot solution volume of 25 mL from a 250 mL stock solution

g Proliss 100 sample	mL of Solution	g carbocysteine	% carbocysteine
0.27	100	.00141	0.52
0.27	200	.000706	0.26
0.27	250	.000565	0.21
0.27	300	.000471	0.17

g Proliss 100 sample	mL of Solution	g carbocysteine	% carbocysteine
0.7530	100	.00141	0.19
0.7530	200	.000706	0.09
0.7530	250	.000565	0.08
0.7530	300	.000471	0.06

g Proliss 100 sample	mL of Solution	g carbocysteine	% carbocysteine
1.02	100	.00141	0.14
1.02	200	.000706	0.07
1.02	250	.000565	0.06
1.02	300	.000471	0.05

Other Possible Calculations:

The purpose is to create a conversion factor called Z for use in QA/QC lab to determine the amount of Proliss 100 in the Keratin Mousse consumer formulation.

Finding conversion Factor Z using the analysis of the 50% Proliss 100 stock..

(mL of iodine titrant)*(percent iodine in titrant)*(aliquot of solution)* (grams of Proliss 100 stock)/mL dilution) Z = 50% Proliss 100 stock

Z = 50%/ [(0.1 mL iodine)*(2 % iodine) * (25 mL aliquot) *(0.27 g Proliss 100 stock/250mL)]

Z = 9260 conversion factor

Use of Z:

need weight of Keratin Mousse sample grams _____g

need dilution volume _____mL

need aliquot volume _____mL

need concentration of iodine titrant _____ %

need mL of iodine titratant _____mL

Percent Proliss in consumer formulations = Z ***(___mL iodine titrant)*(2% iodine titrant)*(25 mL aliquot)***(_____g Consumer product)/(____volume of dilution)

Unfortunately this calculation with this data though it is still not clear on how much % Proliss 100 is in Keratin Mousse:

0.54 g Keratin Mousse

Results and Discussion:

Iodine solution made versus Iodine Solution Provided (Lugol's) yield different product results. It is still unclear whether the % obtained for the Iodine solution made is the amount of Proliss at 50% active or if it's the total amount indicated on formulation production batch sheet made by R&D lab. After clear discussion with R&D manager, the % obtained from iodine solution made was more accurate. The value was the amount of Proliss at 50% active. Results also show that if assume carbocysteine is 100% Proliss and the active ingredient the percent is significantly less than previous % calculated. It unclear as to what is accurate due to not enough information provided from the manufacturing company due to a confidentiality agreement. Unfortunately this method is currently still being perfected for more accuracy and clarity of results as well as perfecting calculations to determine percent Proliss 100 in Keratin mousse sample.

Future Work:

In going forward, suggestions were made to improve the testing of the Proliss. It has been difficult to obtain the molecular weight of the raw material Proliss 100 due to manufacturer's confidentiality agreement. An alternative method involving possibly sodium hydroxide as a titrant and phenalthalein as an indicator could be used as well as a method using sodium thiosulfate as titrant could be considered.

References

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- Certificate of Analysis (COA) from supplier
- MSDS for Proliss 100
- Quantitative Chemical Analysis 7th edition Daniel C. Harris